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(54) METHOD OF CLEANSING POLYESTER-CONTAINING FABRICS

(71) We, THE PROCTER & GAMBLE COMPANY, a Corporation organised and existing under the laws of the State of Ohio, United States of America of 301 East Sixth Street, Cincinnati, Ohio 45202, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

This invention relates to a process for removing oily, i.e. fatty soils and stains, especially triglyceride soils and stains, from a polyester fabric whereby the fabric is soaked in an aqueous solution containing a lipase and calcium and/or magnesium ions and, thereafter, washed in a conventional manner.

Prior-art laundry products and procedures exhibit one or more deficiencies when used to clean oil stains from cloth, especially polyester or polyester fabric blends. Effective removal of triglyceride stains from polyester fabrics has been largely accomplished by relatively inconvenient and expensive methods of dry cleaning. Therefore, a process which would permit the economical and efficient removal of such soils and stains from polyester fabrics and with the aid of conventional household laundry equipment would be desirable.

The present invention provides a process for removing oily soils and stains from a polyester fabric (as hereinafter defined), comprising the steps of (a) soaking the polyester fabric, for a time-period sufficient to degrade the oily soils and stains, in an aqueous soaking solution at a temperature of from 40°F to 160°F and at a pH of from 5 to 11, said solution containing from 1 ppm to 50 ppm of a lipase enzyme having lipolytic activity in the aforesaid temperature and pH ranges, and calcium and/or magnesium ions and (b) thereafter washing said fabric with an aqueous solution of a detergent composition.

The expression "polyester fabric" herein refers not only to fabrics which are entirely of polyester but also to fabrics containing polyester and one or more other materials, especially cotton.

The process of the present invention, whereby oily soils and stains are removed from polyester-containing fabrics, involves two essential steps. The first step consists of soaking the polyester fabrics in an aqueous lipase-containing composition, in the presence of calcium and/or magnesium ions under the conditions described above. During the reaction which proceeds, triglycerides are degraded into diglycerides, monoglycerides and/or glycerine and free fatty acids. The amount of oily soil removed from the fabric depends upon the soaking time period, enzyme concentration, pH of the soak solution, soak temperature, calcium or magnesium ion concentration and the type fabric cleansed.

Lipase is an enzyme which catalyzes the hydrolysis of ester linkages in triglycerides yielding diglycerides, monoglycerides or glycerine and free fatty acids. The hydrolysis of triglycerides by lipase has been known in the art. The use of lipases in cleansing operations has also been known and is described, for example, in German Patent 491,219 (July 16, 1927), British Patent 682,875 (November 19, 1952) and in U.S. Patent 3,451,935 (June 24, 1969). However, the present invention resides in the superior removal of triglyceride stains and soils, e.g. up to 100%, from polyester fabrics or polyester/cotton fabric blends attained by the combined effects of a lipolytic enzyme and calcium and/or magnesium ion. Until the present invention, this level of performance has been available only by dry cleaning the fabric.

The lipases of the invention are those which exhibit lipolytic activity under the conditions of temperature and pH normally encountered in laundry situations. The lipases suitable herein are those which are characterised by sufficient enzymatic activity at a soaking temperature of from

40°F to 160°F in a pH range of from 5 to 11 to degrade and thus render more easily removable the oily soils and stains normally encountered in a laundry situation.

Lipases suitable herein include those of animal, plant or microbiological origin. Few studies have been conducted of lipase distribution in plants, but the enzyme is present in cambium, bark, and in plant roots. In addition, lipases have been shown to be present in the seeds of fruit, oil palm, lettuce, rice bran, barley and malt, wheat, oats and oat flour, cotton, tung kernels, corn, millet, coconuts, walnuts, fusarium, cannabis and cucurbito.

Suitable lipases are also found in many strains of bacteria and fungi. For example, lipases are present in *Pseudomonas*, *Aspergillus*, *Pneumococcus*, *Staphylococcus* and *Staphylococcus toxins*, *Mycobacterium tuberculosis*, *Mycotonia lipolytica*, and *Sclerotinia*.

Suitable animal lipases are found in the body fluids and organs of many species. Most organs of the mammalian body contain lipases, but in addition the enzymes are found in several digestive juices as well as in pancreatic juice. A preferred lipase of animal origin is a pancreatic lipase.

Specific examples of the commercially-available enzymes which are suitable for use herein, the pH ranges of their optimum activity and their source of origin are described as follows:

Lipase	pH Range	Source
Rehzyme PL-600	7-11	Pancreatic Juice
Astra ^o	7-10	Microbial
Nagase	7-9	Microbial
Lipase YL	7-9	Microbial
Wallerstein AW	7-9	Fungal
Amano M-AP	6-8	Fungal
Meito MY-30	6-8	Fungal
Amano CE	6-8	Microbial
Amano CE-50	6-8	Microbial
Amano AP-6	6-8	Fungal
Takeda 1969-4-9 ^o	6-8	Microbial

o "Astra" and "Takeda" are registered trade marks.

The concentration of lipase employed in the soaking step is an amount sufficient under the normal laundry soaking conditions to degrade oily soils and stains so as to effect their removability. While the concentration employed will depend upon the particular enzyme employed, the activity of the enzyme, the nature of the soil or stain, the soaking time, the pH of the soaking solution and the like, a concentration in solution of from: 1 ppm to 50 ppm lipase is employed. The fabrics are usually soaked from 1 to 20 hours preferably from 5 to 10 hours to render polyester and polyester/cotton blends essentially free of triglyceride type stains. The employment of levels of lipase below 1 ppm tends to extend required soaking periods while the use of a concentration of greater than 50 ppm tends to provide little further benefit and is economically unattractive. Swatches of Dacron (a Registered Trade Mark), a polyester fabric, soiled with olive oil and soaked for a four- to five-hour period in a soaking solution containing 50 ppm pancreatic lipase and thereafter washed in a conventional laundry detergent are rendered free of triglyceride soil. A preferred concentration range is from 2 to 10 ppm, the concentration of 10 ppm being particularly desirable for reasons of soil-removability, economic considerations and amount of soak-time necessary for improved soil removal. A 10 ppm solution of lipase will remove over 50% of the triglyceride stains from polyester cloths during a 10-hour soak period; up to 100% of the soil is removed when the soak-time is extended from 10 to 20 hours.

The activity level of the enzyme employed will affect the amount employed in the soaking step. Preferred are enzymes having an activity of from 10,000 to 20,000 units per gram (u/g) as determined by the liberation of fatty acid from a triglyceride substrate under the conditions described more fully in the Report of the Enzyme Committee International Biochemical Union 3-11 (1963).

The lipase can be incorporated into a soaking solution of the invention in a granular or liquid form or in any stable form. Preferably the lipase is employed in a commercially-available form, admixed with inert diluent or vehicle materials, and added to an aqueous bath in an amount sufficient to provide from 1 to 50 ppm of enzyme.

Surfactants can be employed in combination with the lipase and calcium and/or magnesium ion in the soaking step of the process of the invention. Suitable surfactants are those which permit the lipase to exhibit its fat-splitting properties, increase distribution of the enzyme throughout the soaked fabrics and which exhibit cleaning properties. Surfactants which interfere with calcium or magnesium ions are preferably to be avoided. Thus, soaps in amounts which would form lime scum or curds by reaction with all of the hardness ions should be excluded from the soaking step. Inhibition of lipase activity by the action of surfactants tends to be minimised with extended soaking periods.

A wide variety of detergent compounds are suitable for use herein. Examples of detergent surfactants which can be used include but are not limited to:

Synthetic organic detergents characterized by their high solubility in water, their resistance to precipitation by the constituents of hard water and their surface active and effective detergent properties, including:

(a) Anionic synthetic non-soap detergents: This class of synthetic detergents can be broadly described as the water-soluble salts, particularly the alkali metal (sodium, potassium, etc) salts, of organic sulfuric reaction products having in the molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals. Important examples of the synthetic detergents are the sodium or potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols produced by reducing the glycerides of tallow or coconut oil; sodium or potassium alkyl benzene sulfonates, in which the alkyl group in a straight or branched chain contains from about 9 to about 15 carbon atoms; especially those of the types described in United States Letters Patent Nos. 2,220,099 and 2,477,383; sodium alkyl glyceryl ether sulfonates, especially those ethers of the higher alcohols derived from tallow and coconut oil; sodium salts of sulfonated α -olefins containing 8 to 22 carbon atoms, e.g. those described in U.S. patent No. 3,332,850; sodium coconut oil fatty acid monoglyceride sulfates and sulfonates; sodium or potassium salts of sulfuric acid esters of the reaction product of one mole of a higher fatty alcohol (e.g. tallow or coconut oil alcohols) and about three moles of ethylene oxide; sodium or potassium salts of alkyl phenol ethylene oxide ether sulfate with about four units of ethylene oxide per molecule and in which the alkyl radicals contain about 9 carbon atoms; the reaction product of fatty acids esterified with isethionic acid and neutralized with sodium hydroxide where, for example, the fatty acids are derived from coconut oil; sodium or potassium salts of fatty acid amide of a methyl taurine in which the fatty acids, for example, are derived from coconut oil; and others known in the art, a number being specifically set forth in United States Letters Patent Nos. 2,486,921, 2,486,922 and 2,396,278.

(b) Nonionic synthetic detergents: this class of synthetic detergents may be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. The length of the hydrophilic or polyoxyalkylene radical which is condensed with any particular hydrophobic group can be readily adjusted to yield a water-soluble compound having the desired degree of balance between hydrophilic and hydrophobic elements.

For example, a well known class of nonionic synthetic detergents is made available on the market under the trade name of "Pluronic" (Registered Trade Mark). These compounds are formed by condensing ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The hydrophobic portion of the molecule which, of course exhibits water insolubility has a molecular weight of from about 1500 to 1800. The addition of polyoxyethylene radicals to this hydrophobic portion tends to increase the water solubility of the molecule as a whole and the liquid character of the products is retained up to the point where the polyoxyethylene content is about 50% of the total weight of the condensation product.

Other suitable nonionic synthetic detergents include: (i) The polyethylene oxide condensates of alkyl phenols, e.g. the condensation products of alkyl phenols having an alkyl group containing from about 6 to 12 carbon atoms in either a straight chain or branched chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to 10 to 25 moles of ethylene oxide per mole of alkyl phenol. The alkyl substituent in such compounds may be derived from polymerized propylene, diisobutylene, octane, or nonane, for example. (ii) Those derived from the condensation of ethylene oxide with the product resulting from the reaction of propylene oxide and ethylene diamine - products which may be varied in composition depending upon the balance between the hydrophobic and hydrophilic elements which is desired. For example, compounds containing from about 20% to about 80% polyoxyethylene by weight and having a molecular weight of from about 5000 to about 11,000, resulting from the reaction of ethylene oxide groups with a hydrophobic base constituted of the reaction product of ethylene diamine and excess propylene oxide, said base having a molecular weight on the order of 2500 to 3000, are satisfactory. (iii) The condensation product of aliphatic alcohols having from 8 to 18 carbon atoms, in either straight chain or branched chain configuration, with ethylene oxide, e.g. a coconut alcohol ethylene oxide condensate having from 10 to 30 moles of ethylene oxide per mole of coconut alcohol, the coconut alcohol fraction having from 10 to 14 carbon atoms.

(c) Long chain tertiary amine oxides (nonionic detergents) corresponding to the following general formula $R_1R_2R_3N-O$, wherein R_1 contains an alkyl alkenyl or mono-hydroxy alkyl radical of from about 8 to about 18 carbon atoms from 0 to about 10 ethylene oxide moieties, and from 0 to 1 glyceryl moiety, and R_2 and R_3 contain from 1 to about 3 carbon atoms and from 0 to about 1 hydroxy group, e.g. methyl, ethyl, propyl, hydroxy ethyl, or hydroxy propyl radicals. The arrow in the formula is a conventional representation of a semi-polar bond. Examples of amine oxides suitable for use in this invention include dimethyldodecyl amine oxide.

oleyl di(2-hydroxyethyl) amine oxide, dimethyloctylamine oxide, dimethyl-decylamine oxide, dimethyltetradecylamine oxide, 3,6,9-trioxa-heptadecyldiethylamine oxide, di(2-hydroxyethyl) tetradecylamine oxide, 2-dodecoxy ethyl dimethylamine oxide, 3-dodecoxy-2-hydroxy propyl di(3-hydroxypropyl)-amine oxide, dimethyl-hexadecylamine oxide.

(c) Long chain tertiary phosphine oxides (nonionic detergents) corresponding to the following general formula $RR'R''P=O$ wherein R contains an alkyl, alkenyl or mono-hydroxyalkyl radical ranging from 8 to 18 carbon atoms in chain length, from 0 to about 10 ethylene oxide moieties and from 0 to 1 glyceryl moiety and R' and R'' are each alkyl or monohydroxyalkyl groups containing from 1 to 3 carbon atoms. The arrow in the formula is a conventional representation of a semi-polar bond. Examples of suitable phosphine oxides are:

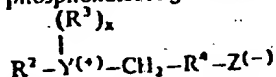
dodecyldimethylphosphine oxide,
tetradecyldimethylphosphine oxide,
tetradecylmethylethylphosphine oxide,
3,6,9-trioxa-octadecyldimethylphosphine oxide,
octyldimethylphosphine oxide,
3-dodecoxy-2-hydroxypropyl di(2-hydroxyethyl) phosphine oxide,
stearyldimethylphosphine oxide,
cetyl ethylpropylphosphine oxide,
oleyl diethylphosphine oxide,
dodecyldiethylphosphine oxide,
tetradecyldiethylphosphine oxide,
dodecyldipropylphosphine oxide,
dodecyldi(hydroxymethyl) phosphine oxide,
dodecyldi(2-hydroxyethyl) phosphine oxide,
tetradecylmethyl-2-hydroxypropyl phosphine oxide,
oleyldimethylphosphine oxide, and
2-hydroxydodecyldimethylphosphine oxide.

(e) Long chain dialkyl sulfoxides containing one short chain alkyl or hydroxy alkyl radical of 1 to about 3 carbon atoms (usually methyl) and one long hydrophobic chain which contains alkyl, alkenyl, hydroxy alkyl, or keto alkyl radicals containing from about 8 to about 20 carbon atoms, from 0 to about 10 ethylene oxide moieties and from 0 to 1 glyceryl moiety. Examples include:

octadecyl methyl sulfoxide,
2-ketotridecyl methyl sulfoxide,
3,6,9-trioxa-octadecyl 2-hydroxyethyl sulfoxide,
dodecyl methyl sulfoxide,
oleyl 3-hydroxy propyl sulfoxide,
tetradecyl methyl sulfoxide,
3-methoxytridecyl methyl sulfoxide,
3-hydroxytridecyl methyl sulfoxide,
3-hydroxy-4-dodecoxybutyl methyl sulfoxide.

(f) Ampholytic synthetic detergents can be broadly described as derivatives of aliphatic secondary and tertiary amines in which the aliphatic radical can be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water solubilising group, e.g. carboxy, sulfonate, sulfate, phosphate, or phosphonate. Examples of compounds falling within this definition are sodium 3-dodecylaminopropionate, sodium 3-dodecylaminopropane sulfonate, dodecyl-beta-alanine, N-alkyl-taurines such as the one prepared by reacting dodecylamine with sodium isethionate according to the teaching of U.S. 2,658,072, N-larger alkyl aspartic acids such as those produced according to the teaching of U.S. 2,438,091, and the products sold under the trade name "Miranol" and described in U.S. Patent 2,528,378.

(g) Zwitterionic synthetic detergents can be broadly described as derivatives of aliphatic quaternary ammonium, phosphonium, and sulfonium compounds, in which the aliphatic radicals can be straight chain or branched, and wherein one of the aliphatic substituents contains from about 8 to 18 carbon atoms and one contains an anionic water solubilising group, e.g. carboxy, sulfonate, sulfate, phosphate, or phosphonate. A general formula for these compounds is:



wherein R² contains an alkyl, alkenyl, or hydroxy alkyl radical of from about 8 to about 18 carbon atoms, from 0 to about 10 ethylene oxide moieties and from 0 to 1 glyceryl moiety; Y is selected from the group consisting of nitrogen, phosphorous, and sulfur atoms; R⁴ is an alkyl or monohydroxy alkyl group containing 1 to about 3 carbon atoms; x is 1 when y is a sulfur atom and 2 when y is a nitrogen or phosphorous atom, R⁴ is an alkylene or hydroxy alkylene of from 1 to about 4 carbon atoms and Z is a radical selected from the group consisting of carboxylate, sulfonate, sulfate, phosphonate, and phosphate groups.

Examples include 3-(N,N-dimethyl-N-hexadecylammonio) propane-1-sulfonate and 3-(N,N-dimethyl-N-hexadecylammonio)-2-hydroxypropane-1-sulfonate which are especially preferred for their excellent cool water detergency characteristics.

The alkyl groups contained in said detergent surfactants can be straight or branched preferably straight and saturated or unsaturated as desired. The above list of detergent surfactants is exemplary and not limiting. Mixtures of the above detergent surfactants can be used.

The lipase and calcium or magnesium salt which provides a source of hardness ion can be formulated, optionally in combination with surfactants described hereinbefore, certain builders and enzyme stabilisers in any suitable physical form such as granules (e.g. either spray-dried or mechanically mixed), tablets, pastes or liquids. Such compositions can be added to water to provide an aqueous soaking bath for treatment of fabrics as described hereinbefore.

The compositions can also contain particulate inorganic salts which are inert, provide an electrolyte content and which act as fillers. Examples of such salts include sodium sulfate and sodium chloride.

Sequestering builder salts which gather or otherwise prevent the calcium or magnesium ions from exhibiting their beneficial effects in soaking solution should be avoided. Thus, polyphosphates, aminopolycarboxylates and other sequestrants are preferably avoided. Inorganic alkaline non-sequestering builder salts such as sodium sesquicarbonate, sodium tetraborate, sodium orthophosphate, the corresponding potassium salts and mixtures thereof can be suitably employed. Accordingly, the soaking step of the process of the invention, in addition to being conducted in the absence of interfering amounts of soap, is effected in the absence of amounts of sequestering builder salts sufficient to remove substantially all the calcium or magnesium ions from the solution.

Compositions containing a detergent non-soap surfactant and a non-sequestering builder salt will normally contain these components in amounts corresponding to a ratio of such builder salt to surfactant of from 1:1 to 10:1, and preferably 1:1 to 5:1.

The pH of the soaking step can be maintained at a particular and desired level by the incorporation of a buffering agent. While the soaking step will be effected at a pH of from 5 to 11, buffering agents can be employed to regulate pH within a desired range, e.g. the preferred range of 8 to 10, so as to maximise the activity of the lipase employed. Preferred buffers for use herein are sodium tetraborate decahydrate and boric acid which maintain an acid-base balance within a pH range corresponding to the optimum activity level. Other suitable buffers can be employed to maintain a desired pH range depending on the particular lipase used in the presoak. Examples of other buffers include sodium bisulfate, potassium bisulfate, monosodium phosphate, and sodium acid pyrophosphates or mixtures thereof.

Other minor ingredients can also be present in the soaking compositions. Soil-suspending agents such as sodium carboxymethylcellulose, optical brighteners, dyes, germicidal agents, suds depressants, and suds boosters, can each be added in amounts up to about 10% by weight of the composition.

An essential aspect of the soaking step of the invention is that the soaking be effected in the presence of calcium and/or magnesium ions. It has been discovered that the presence of a source of such hardness ions with the lipase in the soaking step greatly facilitates the removal of fatty soils and stains from polyester fabrics. The same removal is not observed in connection with the soaking of cotton goods having oily soils and stains. The effect of calcium ion on lipase stability has been described in the art, for example, by E.D. Wills, in *Advance in Lipid Research* 3, 197 to 240 (1965); the effect of hardness on lipase triglyceride-soil-removal from polyester fabrics, however, is not described. While applicant does not wish to be bound by any theory as to the mechanism involved whereby calcium or magnesium ion plays a role in stain-removal, particularly in the case of polyester fabrics, penetration of oil into the lumen (hollow circular cavity) of cotton fibres and inaccessibility of the oil to the action of the enzyme may be involved.

The amount of calcium or magnesium hardness ion employed in the soaking step will vary depending upon the soaking pH and temperature and type of lipase used. For example, complete, i.e. 100% removal of fatty soil can be obtained using a minimum concentration of about 1.5 gr/gal. (i.e. 2.42×10^{-4} M) hardness ion with 50 ppm Rehyzyme PL-600 at a pH of 10 and a temperature of 80°F and for a soaking time of from about 10 to 20 hours. (The term "gallon" or "gal." herein refers to the U.S. gallon). At lower pH values (e.g. pH of 8), complete soil removal is accomplished when a concentration of at least about 7 gr/gal. (1.22×10^{-3} M) hardness ion is employed under the above-described soaking conditions. Preferably from 6 to 20 grains/gallon calcium or magnesium ion, especially 10 to 15, is employed to assure high level soil removal performance at pH conditions of 6 to 8.

Normally, ordinary tap water will contain dissolved minerals such as calcium and magnesium ions in amounts that will vary with geographical location. Thus, the natural water hardness of varying water supplies may or may not be sufficient to provide the level of performance attainable in the exercise of the present invention.

Accordingly, the calcium and/or magnesium ions may be provided by a water-soluble calcium

or magnesium salt, preferably selected from calcium nitrate, calcium chloride, calcium sulfate, magnesium nitrate, magnesium chloride, magnesium sulfate and mixtures thereof. Hardness of the soak and/or wash water is found to enhance rather than inhibit the lipase hydrolysis of oily stains.

The soaking step of the process of the present invention can be carried out in any of the types of containers normally found in the household. Thus, the soaking step can be conducted in basins, tubs, washtubs, buckets, pails or the like so as to effect the soil- and stain-removing function of the combined employment of lipase and calcium and/or magnesium ion. A preferred method of effecting the soaking operation involves the use of the laundry tub or basin of a conventional home washing machine. The conduct of the soaking step in this fashion eliminates the need for removing the garments for a subsequent washing operation which will normally be effected in a washing machine. The soaking of the fabrics can be conducted with the aid of agitation. Such agitation is conveniently employed as a means of reducing the soaking time required to effect soil and stain removal.

The soaking operation can be conducted in accordance with usual laundry soaking methods. A temperature in the range of from 40°F to 160°F is used in order to ensure substantial enzymatic activity. A preferred temperature is from 90°F to 130°F.

The washing step, which follows the pre-soaking step described hereinbefore, is conducted in the presence of an additional organic detergent composition. Suitable detergents for employment in the washing step include, for example, any of the commercially-available heavy-duty laundry compositions employed in home laundering. Such detergent compositions will comprise a water-soluble organic detergent and, preferably, a builder salt for enhanced cleaning properties. Examples of suitable detergent compounds which can be employed are those described hereinbefore and the alkali metal soaps, such as the sodium and potassium salts, of naturally occurring plant or animal esters (e.g. palm oil, coconut oil, babassu oil, soybean oil, castor oil, tallow, whale and fish oils, grease and lard, and mixtures thereof) or of synthetically produced fatty acids (e.g. rosin and those resin acids in tall oil) and/or of naphthenic acids.

Suitable builder salts will include alkali metal carbonates, borates, phosphates, polyphosphates, bicarbonates and silicates. (Ammonium or substituted ammonium salts can also be used). Specific examples of such salts are sodium tripolyphosphate, sodium carbonate, sodium tetraborate, sodium pyrophosphate, sodium bicarbonate, potassium tripolyphosphate, sodium hexamethaphosphate, sodium sesquicarbonate, sodium mono- and diortho phosphate and potassium bicarbonate.

Examples of organic alkaline sequestant builder salts used alone or in admixture include alkali metal, ammonium or substituted ammonium, aminopolycarboxylates, e.g. sodium and potassium N-(2-hydroxyethyl)-ethylenediaminetriacetates, sodium and potassium nitrilotriacetates and sodium, potassium and triethanolammonium N-(2-hydroxyethyl)-nitrile diacetates. Mixed salts of these polycarboxylates are also suitable. The alkali metal salts of phytic acid, e.g. sodium phytate are also suitable as organic alkaline sequestant builder salts (see U.S. Patent 2,739,942). The detergent and builder components will normally be employed in such amounts as to provide a ratio of builder salt to organic detergent of from about 1:1 to 10:1.

Following completion of the lipase pre-soaking operation of the invention, the treated fabrics are laundered in a conventional manner with the aid of an additional laundry composition comprising detergent surfactant and preferably builder components as described. The laundry detergent component can be added into the aqueous presoaking bath so as to conveniently effect the two-step process of the present invention. It is usual to remove the bulk of the soak liquor and it may be desirable to provide a rinsing operation subsequent to the lipase presoaking step and prior to the detergent washing operation. Such steps minimize the effect of the calcium and/or magnesium ions of the presoaking step on the level of performance attainable from the subsequent laundry washing step. The detergent washing step, which involves the use of conventional laundry detergent compositions in amounts customarily employed in home laundering operations, facilitates the removal of the fragmented products resulting from the fat-splitting properties of the lipase employed in the soaking step. Thus, the hydrolyzed products of the soaking, which may include glycerine and fatty acids, are removed from the treated fabrics to provide improved cleaning effects.

Fabrics treated in accordance with the presoaking and washing steps of the present invention are rinsed in the usual fashion. The rinsing operation can be conveniently effected in a home washing machine as the usual rinsing operation which follows a conventional detergent wash. Following rinsing, the treated fabrics can be dried, ironed or folded as is customary in the laundry arts.

The process of the present invention finds applicability to the removal of oily soils and stains from a number of polyester or polyester-containing fabric materials. Thus, the process can be employed to remove oily soils and stains from fabrics woven from polyester fibres or from fabrics which employ combinations of polyester fibres and other fibres (e.g. 65% polyester, 35% cotton blends). Examples of polyester fabrics are those woven or nonwoven materials fabricated from polyester fibres, the most common of which are copolymers of ethylene glycol and terephthalic

acid. Such fabrics are commercially available under a number of trade names, e.g. Dacron, Fortrel (Registered Trade Mark), Kodol (Registered Trade Mark) and Blue C. Polyester.

In the Examples which follow, cotton, polyester and polyester/cotton blends were used to determine the effect of a lipase/hardness ion (Ca^{++} and Mg^{++}) presoak on triglyceride stains. Swatches were prepared from cotton, Dacron, polyester (Fortrel)/cotton blends; polyester (Fortrel)/cotton blends having a durable press (DP) finish; and polyester (Dacron)/cotton blends having durable press and soil release (SR) finishes. The swatches were soiled, soaked and washed in accordance with procedures described hereinafter.

Triglyceride stains were imparted to the fabrics by one of two methods. Olive oil-type stains (e.g. olive oil, triolein) were applied by dipping the fabric into a solution containing the triglyceride compound. Olive oil was diluted with isobutyl alcohol so that an even, constant soil level was achieved. The soil level was adjusted by varying the oil to alcohol ratio. The various fabrics were then dipped into the soiling solution and the alcohol was allowed to evaporate. Before soiling, all of the swatches were placed in a constant temperature and humidity room for a period of 24 hours and weighed. After soiling, the alcohol on the swatches was allowed to evaporate and the fabrics were reweighed to obtain the soil weight.

Kraft (Registered Trade Mark) French dressing type soils were applied to the fabric by use of an eye dropper; the fabric was saturated with the oil and the excess dressing was allowed to absorb onto an absorbent material. The stains were contained in a circle of about 1-1/2 inches in diameter. A good duplication of soil level was maintained in this manner. The weighing was conducted utilising the same method used for olive oil stains.

Soaking was conducted by soaking each soiled swatch in a separate beaker containing 100 ml. of the soaking solution. Soaking solutions were provided by preparing a borate buffer solution containing 6.07 g $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ and 2.33 g H_3BO_3 per litre of distilled water, adjusting to desired and specified pH with hydrochloric acid or sodium hydroxide as required and adding as calcium chloride dihydrate and magnesium chloride hexahydrate ($\text{Ca}^{++}/\text{Mg}^{++}$ weight/ratio 3/1) to a water hardness of 6 grains/gallon. Lipase (Rehyzyme PL-600, a pancreatic lipase from the pancreas of pigs) was then added at the specified concentration prior to soaking of the swatches. The swatches were rinsed after the soaking step to remove any scum which may have been deposited upon the fabrics.

The soaking and rinsing steps were followed in each Example by a washing operation, which was conducted for 10 minutes at 130°F followed by two rinses at two minutes each. The detergent composition employed consisted of 20% (by weight) C_{11-18} linear alkyl sulfonate, 50% sodium tripolyphosphate, 24% sodium sulfate and 6% sodium silicate. The detergent composition was 0.12% by weight of the aqueous wash solution. All washing and subsequent rinsing steps were conducted in a miniature washing machine (Tergotometer) designed to simulate a conventional washing machine. The percent soil removal was calculated on the basis of weighing made before soiling, after soiling and after the washing step.

Example I

Cotton, Dacron and polyester fabric/blends were soiled with olive oil (8% level) and allowed to soak for 20 hours at 100°F in a solution containing 50 ppm Rehyzyme PL-600 lipase; 6 grains/gallon hardness ion in the $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, H_3BO_3 buffer solution hereinbefore described; and at pH 10. The swatches were analysed gravimetrically both before and after soiling to obtain the soil weight. The weighing procedure was repeated after the lipase presoak and detergent wash to obtain the percent soil hydrolysed from the swatch. Results of the soaking and washing method of the invention are set forth in Table I.

Table I

Fabric	% Soil Removal (by weight)
100% cotton (control)	33%
25/75 polyester/cotton	93%
25/75 polyester/cotton + DP	98%
35/65 polyester/cotton + SR	72%
35/65 polyester/cotton + SR + DP	88%
65/35 polyester/cotton	83%
65/35 polyester/cotton + DP	90%
65/35 polyester/cotton + DP + SR	100%
100% Dacron	97%

DP = Durable press finish SR = soil release finish

As is apparent from Table I, the employment of a presoaking operation (6 gr./gallon hardness; 20 hours; 100°F; pH 10; 50 ppm Rehyzyme PL 600 and buffered with $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, H_3BO_3) followed by a detergent wash effects significant removal of olive oil stains from synthetic polyester

and polyester/cotton blend fabrics. The removal is in contrast to that obtained in the case of the cotton control.

Example II

Dacron swatches were soiled with triolein (8%) and soaked and washed in accordance with the procedures of Example I. Soaking conditions were: pH 10; 20 hours; 100°F; 6 gr/gallon hardness; $\text{Na}_2\text{B}_4\text{O}_7$, H_3BO_3 buffer. The lipase employed (Rehzyme PL-600) was employed in the amounts shown in Table II.

Table II

Lipase Concentration	% Soil Removal
0 ppm	14%
1 ppm	47%
10 ppm	91%
50 ppm	96%

In each case the lipase presoak enhanced the removal of triglyceride stains.

Example III

Dacron was soiled at the 62% by weight level with French dressing (Kraft) employing the soiling method described hereinbefore. The swatch was soaked, washed and percent soil removal was determined. Soaking conditions were pH 10, 20 hours; 100°F; 6 gr/gallon hardness; $\text{Na}_2\text{B}_4\text{O}_7$, H_3BO_3 buffer; 50 ppm Rehzyme PL-600. Soil removal of 75% was effected.

Example IV

Dacron swatches soiled with olive oil at an 8% level were treated in accordance with the procedures of Example I. Soaking conditions were: pH 8; 20 hours; 80°C; $\text{Na}_2\text{B}_4\text{O}_7$, H_3BO_3 buffer; 50 ppm Rehzyme PL-600. The amount of hardness ion present is indicated in Table III along with soil removal results. Data in excess of 100% represent analytical error.

Table III

Hardness (gr./gal.)	Soil removal (%)
0	39
0.5	61
1	73
2	78
5	93
10	103
20	102

In the specification of our copending patent application No. 57771/73 (Serial No. 1442419) there is described and claimed a process for removing oily soils and stains from a polyester fabric (as hereinbefore defined), comprising the steps of (1) soaking the fabric in an aqueous solution at a temperature of from 40°F to 160°F and at a pH of from 6 to 11, said solution containing from 1 ppm to 100 ppm of a lipase enzyme having activity in the aforesaid temperature and pH ranges and at least 50 ppm of a borax salt of the formula $\text{M}_2\text{B}_4\text{O}_7 \cdot x\text{H}_2\text{O}$, wherein M is an alkali metal cation and wherein x is an integer of from 6 to 11, for a period of at least one hour, and (2) thereafter washing said fabric with an aqueous solution of a detergent composition.

WHAT WE CLAIM IS:

1. A process for removing oily soils and stains from a polyester fabric (as hereinbefore defined), comprising the steps of (a) soaking the polyester fabric, for a time-period sufficient to degrade the oily soils and stains, in an aqueous soaking solution at a temperature of from 40°F to 160°F and at a pH of from 5 to 11, said solution containing from 1 ppm to 50 ppm of a lipase enzyme having lipolytic activity in the aforesaid temperature and pH ranges, and calcium and/or magnesium ions and (b) thereafter washing said fabric with an aqueous solution of a detergent composition.

2. A process according to claim 1 wherein the lipase enzyme is of animal, plant or microbiological origin.

3. A process according to claim 1 or 2 wherein the activity of the lipase is from 10,000 to 20,000 units/g.

4. A process according to any one of claims 1 to 3 wherein the calcium and/or magnesium salt concentration in the aqueous soaking bath is from 6 grains/gallon to 20 grains/gallon.

5. A process according to claim 4, wherein the said salt concentration is from 10 grains/gallon to 15 grains/gallon.

6. A process according to any one of claims 1 to 5, wherein the calcium and/or magnesium ions are provided from a water-soluble salt selected from calcium nitrate, calcium chloride, calcium sulfate, magnesium nitrate, magnesium chloride, magnesium sulfate, and mixtures thereof.
7. A process according to any of claims 1 to 6, wherein the fabric is soaked at a temperature of from 90°F to 130°F.
8. A process according to any one of claims 1 to 7, wherein the soaking is effected for a period of from 1 to 20 hours.
9. A process according to claim 8, wherein the soaking is effected for a period of from 5 to 10 hours.
10. A process according to any one of claims 1 to 9, wherein the concentration of the lipase is from 2 to 10 ppm.
11. A process according to any of claims 1 to 10, wherein the said pH is from 8 to 10.
12. A process according to any one of claims 1 to 11, wherein the soaking step is effected in the substantial absence of soaps and of sequestering agents.
13. A process according to any one of claims 1 to 12, wherein the polyester of the fabric is a copolymer of terephthalic acid and ethylene glycol.
14. A process according to claim 1 substantially as described in any one of the Examples.
15. A polyester fabric having freed of oily soils and stains by the method to any one of claims 1 to 14.

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